Performance Monitoring Protocol (QA/QC) for the JEOL AccuTOF DART

1 Scope

This document addresses the performance monitoring (QA/QC) of the JEOL AccuTOF DART. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, paint, explosives (chemistry), and Chemistry Unit general physical and chemical analysis.

2 Principle

This system consists of a JEOL AccuTOF Time-of-Flight (TOF) mass spectrometer (MS) and an IonSense Direct Analysis in Real Time Source (DART) ionization source. The TOF has the ability to measure mass accuracy with less than 5 milli-mass units (mmu) error over a range of 1 - 10,000 m/z. The DART source provides the capability of direct ionization with little to no sample preparation needed. The combined system may also be referred to as a 'DART.'

The TOF tube is sensitive to internal and external changes in temperature and humidity. These changes may result in a uniform shift of mass assignments. This shift is easily corrected by analyzing the calibrant in every sample analysis/data file and applying the new calibration algorithm, producing mass-corrected accurate mass data with less than 5 mmu error.

This performance monitoring protocol is based upon the manufacturer’s recommendations. It should be noted that the instrument manufacturer can be referred to as either JEOL or IonSense. DART instrumentation and supplies are sold and supported by JEOL USA, Inc. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol" and the "Mass Spectrometer General Maintenance Protocol".

3 Equipment/Materials/Reagents

a. Instrumentation - JEOL AccuTOF MS, IonSense DART Source, and data system with IonSense DART Controller and MassCenter Main software (or equivalent).

b. Source Gas - Helium, 99.99% or greater purity (or equivalent)

c. Source Gas - Nitrogen, 99.99% or greater purity (or equivalent)

d. Capillary tubes

e. Polyethylene Glycol (PEG), average molecular weight of 550 (Sigma or equivalent)

4 Standards and Controls

Neat Polyethylene Glycol (PEG) is used for performance verification and mass correction.

5 Calibration

The PEG is analyzed with every sample in the same data file, and used for fine mass correction. This results in the highest mass accuracy possible, taking all instrumental and environmental conditions into account at the time of analysis. This also ensures that calibration data will be available at any time in the future when data processing is performed. The operator is responsible for verifying that every calibration graph produced passes the 'Decision Criteria' section of this SOP. However, only the first calibration graph is printed and recorded.

6 Sampling or Sample Selection

Not applicable.

7 Procedures

7.1 Start Up

7.1.1 DART Initialization

a. Open the DART Controller Software.

b. Select ‘Standby’.

c. Select the appropriate operating temperature and gas type.

d. Set the polarity required.

e. Once DART has reached the operating temperature, select ‘Run’.

7.1.2 TOF Initialization

a. Open Mass Center Main.

b. Open MS Tune Manager.

c. Put instrument into 'Operate' mode.

d. Load the appropriate tune file, verifying that the polarity matches the DART source.

e. Verify that the voltage for Orifice 1 is set to 30 V (positive for positive ion mode,

negative for negative ion mode).

f. Verify that the appropriate analyzer settings.

7.2 Daily Checks

a. Record the remaining disk space on the hard drive. Use Windows Explorer program to verify that the hard disk has at least 20 GB of free disk space. Do not use if less than 20 GB remain. If less than 20 GB of free disk space remain, contact appropriate instrument support personnel.

b. Record the line pressure of the building nitrogen supply. The regulator should read at least 70 p.s.i. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the nitrogen is supplied by a gas cylinder, record the tank volume pressure. Change the tank if less than 100 p.s.i. remaining.

c. Record the line pressure of the helium. The regulator should read at least 40 p.s.i. If it cannot maintain this pressure, contact appropriate instrument support personnel. If the helium is supplied by a gas cylinder, record the tank volume pressure. Change the tank if less than 100 p.s.i. remaining.

d. Check the vacuum pressure readings. The analyzer pressure must be below 9.9 x 10^{-5} Pa with the DART controller in standby (i.e., nitrogen flowing, not the helium).

e. Perform a data acquisition for PEG using the procedure in 7.3 below. A PEG analysis from a sample acquisition collected the same day can be used as well.

7.3 Data Acquisition

a. Open Spectrum Monitor. Ensure that there is a signal.

b. Start an acquisition by selecting 'Acquire.' Type in the filename and comments and choose a data folder. Set the desired m/z range and verify the length of acquisition is appropriate (typically 2 minutes).

c. Once the acquisition has started, ensure that there is a signal, and collect several seconds of background data.

d. Dip the closed end of a glass capillary into the PEG, then place in the DART gas stream until a response is seen in the spectrum viewer for several seconds. Do this at least twice during the acquisition.

7.3.1 Time of Use Performance Verification/Mass Correction

Verify the performance of the instrument in the first data file acquired:

a. Open the sample data file in the Chromatogram Viewer.

b. View a background-subtracted PEG spectrum by holding down the 'shift' key and dragging across the PEG area with the right mouse button, then drag across a baseline area while holding the 'ctrl' key.

c. Once the PEG spectrum appears in the Spectrum Viewer, generate a centroided spectrum.

d. Right mouse click on the centroided spectrum, and make a calibration file from the spectrum.

e. Select the appropriate polynomial order to provide the lowest residuals curve (1-R and $1\text{-}R^{*}$ less than 10^{-10}).

f. Evaluate the results using the 'Decision Criteria' section of this protocol.

g. If the results are acceptable, print the PEG calibration graph.

h. Save the calibration using an appropriate file name. The calibration file can now be applied to the sample chromatogram to obtain accurate mass data.

7.3.2 Data Correction

Verify the performance of the instrument in every data file acquired thereafter.

a. The steps listed under 7.3.1 will be followed for every sample acquisition in order to ensure accurate mass results. However, it is not necessary to record subsequent results in the QA/QC log for the same day.

b. Verify that the residuals (1-R and 1-R*) are 10^{-10} or lower before applying the calibration algorithm to the sample.

7.4 Instrument Shutdown

7.4.1 Time of Use Shutdown

a. Put the MS Tune Manager in ‘Analyzer HV’ mode.

b. Put the DART in 'Standby' mode, which will maintain the voltages and switch to nitrogen as the flow gas.

c. Turn the heater off with nitrogen flowing to allow the heater to cool.

7.4.2 End of Day Shutdown

a. Put the MS Tune Manager in ‘Analyzer HV’ mode.

b. Turn the DART off, which will turn off the voltages and turn off the nitrogen flow gas.

7.4.3 As Needed Maintenance

a. Replace the grid.

b. Clean the cones, source, and source enclosure.

8 Instrumental Conditions

8.1 Mass Spectrometer

Ionization:	DART
Scan Mode:	Profile mode
Analyzer:	500 V peaks voltage for 50 - 500 m/z 800 V peaks voltage for 80 – 800 m/z

9 Decision Criteria

9.1 PEG Performance Verification/Calibration

Verify that the residuals (1-R and 1-R*) are 10^{-10} or lower.

10 Calculations

Not applicable.

11 Measurement Uncertainty

Not applicable.

12 Limitations

Not applicable.

13 Safety

There are several areas of the MS which utilize extremely high voltage and vacuum conditions. For this reason, maintenance should only be performed with the system vented and the main

power off. Many MS components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis.

14 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual.*

"Liquid Chromatograph General Maintenance Protocol" (Inst 003) *Instrument Operation and Systems Support SOP Manual.*

"Mass Spectrometer General Maintenance Protocol" (Inst 004) *Instrument Operation and Systems Support SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
1	09/21/09	Removed hyphen in AccuTOF in title and sections 1 and 3a. Removed the word 'unique' from description of DART in section 2. In section 7.1.1c and d, changed heater temperature and needle voltage to values commonly used. Changed section 7.1.2e and f to clarify voltage polarity, and added the peaks voltage. Changed section 7.2a to increase the size of the hard drive disk space minimum. Re-worded section 7.2d to further clarify vacuum reading conditions. Changed section 7.3b to a fixed mass range. Re-worded section 7.3d to specify only PEG standard. Changed section 7.4.2b to more accurately describe the DART shutdown procedure. Added peaks voltage to section 8.1.
2	10/04/18	Updated Section 1 Scope to include disciplines/categories of testing. Updated Sections 7.1.1, 7.3.1, 7.4.1 and 7.4.2 for changes in latest software. Changed to ‘appropriate analyzer settings’ in Section 7.1.2 f. Added ‘appropriate instrument support personnel’ to Section 7.2 a, b & c. Added more detail to Section 7.3 d. Updated ‘Instrument Operation and Systems Support’ in Section 14 and header.

Approval

Redacted - Signatures on File

Drug Chemistry/
General Chemistry
Technical Leader: Date: 09/28/2018

Toxicology
Technical Leader: Date: 09/28/2018

Paints and Polymers
Technical Leader: Date: 09/28/2018

Explosives (Chemistry)
Technical Leader: Date: 09/28/2018

IOSS Manager: Date: 09/28/2018

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Chemistry Unit Chief: Date: 09/28/2018

QA Approval

Quality Manager: Date: 09/28/2018